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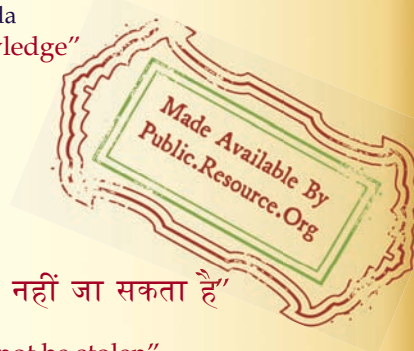
IS 4321 (1989): Pesticides - 2, 4-D, Technical [FAD 1: Pesticides and Pesticides Residue Analysis]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS 4321 : 1989
(Reaffirmed 1999)
Edition 3.1
(1994-09)

Indian Standard

PESTICIDES — 2, 4-D, TECHNICAL —
SPECIFICATION

(Second Revision)

(Incorporating Amendment No. 1)

UDC 632.954

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Price Group 2

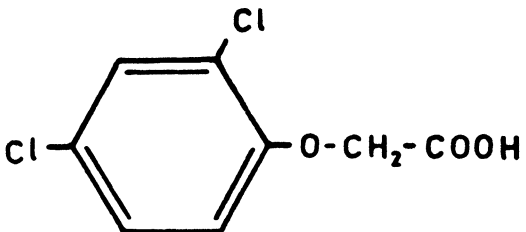
FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards on 24 April 1989, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council.

2, 4-D, technical is employed in the preparation of herbicidal formulations used for the control of weeds.

2, 4-D, technical is generally manufactured to contain 97 percent (*m/m*) of 2, 4-dichlorophenoxyacetic acid (2, 4-D).

2, 4-D is the accepted common name by the International Organization for Standardization (ISO) for 2, 4-dichlorophenoxyacetic acid. The empirical and structural formulae and the molecular mass are as indicated below:

<i>Empirical Formula</i>	<i>Structural Formula</i>	<i>Molecular Mass</i>
$C_8H_6Cl_2O_3$		221.0

In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act, 1968* and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

This edition 3.1 incorporates Amendment No. 1 (September 1994). Side bar indicates modification of the text as the result of incorporation of the amendment.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

PESTICIDES — 2, 4-D, TECHNICAL — SPECIFICATION

(Second Revision)

1 SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for 2, 4-D technical.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	Title
IS 1070 : 1977	Water for general laboratory use (<i>second revision</i>)
IS 1488 : 1989	Pesticides — 2, 4-D sodium salt, technical — Specification
IS 6940 : 1982	Methods of test for pesticides and their formulations (<i>first revision</i>)
IS 8190 (Part 1): 1988	Requirements for packing of pesticides : Part 1 Solid pesticides (<i>second revision</i>)
IS 10946 : 1984	Methods of sampling for technical grade pesticides

3 REQUIREMENTS

3.1 Description

It shall be white to light coloured powder with slight phenolic odour.

3.2 Identity Test

The *R_f* value of the material shall be the same as that of standard 2, 4-D when tested by the method prescribed in IS 1488 : 1989. Sample of the material shall be used instead of extracted 2, 4-D.

3.3 The material shall also comply with the requirements given in Table 1.

4 PACKING

4.1 The material shall be packed according to IS 8190 (Part 1) : 1988.

5 MARKING

5.1 The following information shall be marked legibly and indelibly on each container in addition to any other information required under the *Insecticides Act, 1968* and Rules:

- Name of the material;
- Name of the manufacturer;
- Date of manufacture;

- Batch number;
- Nominal 2, 4-D content, percent (*m/m*);
- Net mass of contents; and
- The minimum cautionary notice as worded in *Insecticides Act, 1968* and Rules.

Table 1 Requirements for 2, 4-D Technical
(*Clauses 3.3 and 7.1*)

Sl No.	Characteristic	Require- ment	Method of Test, Ref to	
			Annex of This Stan- dard	Cl No. of IS 6940 : 1982
(1)	(2)	(3)	(4)	(5)
i)	Acid content (calculated as 2, 4-D), percent by mass, <i>Min</i>	97.1	A	—
ii)	Free phenol (calculated as 2, 4-dichlorophenol), percent by mass, <i>Max</i>	1.0	B	—
iii)	Loss on drying at 105°C, percent by mass, <i>Max</i>	0.5	C	—
iv)	Melting point, °C	136 to 140	—	6
v)	Triethanolamine insoluble matter, percent by mass, <i>Max</i>	0.5	D	—
vi)	Sulphated ash, percent by mass, <i>Max</i>	1.0	E	—

6 SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in IS 10946 : 1984.

7 TESTS

7.1 Tests shall be carried out by the appropriate methods referred to in col 4 and 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[Table 1, Item (i)]

DETERMINATION OF ACID CONTENT

A-1 GENERAL

A-1.1 The 2, 4-D content of the technical material is determined by the procedure as given in **A-3** and taking into consideration the value obtained as equivalent mass of the material taken.

A-2 REAGENTS

A-2.1 Ethyl Alcohol

Neutral, alternatively methyl alcohol, neutral may be used.

A-2.2 Standard Sodium Hydroxide Solution, 1 N.

A-2.3 Bromothymol Blue Indicator Solution, 0.04 percent solution in alcohol (*m/v*).

A-3 PROCEDURE

A-3.1 Weigh accurately about 5 g of the sample. Wash it into a 250-ml conical flask with 50 ml

of neutral alcohol. Shake to dissolve the material and dilute it with 50 ml of distilled water. Titrate with standard sodium hydroxide solution using bromothymol blue as indicator to a green colour end point.

A-4 CALCULATION

A-4.1 Acid content (calculated as 2, 4-D, equivalent mass being 221.0), percent by mass = $\frac{V \times N \times 22.1}{M}$

where

V = volume, in ml, of the standard sodium hydroxide solution;

N = normality of the standard sodium hydroxide solution; and

M = mass, in g, of the material taken for the test.

ANNEX B

[Table 1, Item (ii)]

DETERMINATION OF FREE PHENOLS IN 2, 4-D

B-1 OUTLINE OF THE PROCEDURE

B-1.1 The absorbance of acetone-ammonia solution of the acid is determined after adding 4-aminophenazone and potassium ferricyanide solution and comparing the colour development with the similarly prepared solution of standard 2, 4-dichlorophenol solution.

B-2 REAGENTS

B-2.1 Standard 2, 4-Dichlorophenol Solution

Prepare by dissolving 100 mg of pure 2, 4-dichlorophenol in 10 ml of acetone and diluting to 500 ml with distilled water. Dilute 10 ml of the resulting solution to 100 ml with distilled water. One millilitre of the final solution is equivalent to 20 µg of 2, 4-dichlorophenol.

B-2.2 Standard 4-Aminophenazone Solution — 2 percent (*m/v*), in distilled water and to be stored in dark.

B-2.3 Standard Potassium Ferricyanide Solution — 8 percent (*m/v*), freshly prepared in distilled water.

B-2.4 Standard Ammonium Hydroxide Solution — 0.1N.

B-3 PROCEDURE AND CALIBRATION

B-3.1 Dissolve 200 mg of the sample in 60 ml of Standard ammonium hydroxide solution, dilute to 500 ml with distilled water, and take with a pipette 50 ml of the resulting solution into a conical flask. Add 0.5 ml of the standard 4-aminophenazone solution, shake, add 0.25 ml of the potassium ferricyanide solution, and shake again for one minute.

B-3.2 Pipette 10 ml of the 2, 4-dichlorophenol standard solution into a conical flask, add 5 ml of standard ammonium hydroxide solution and dilute to 50 ml with distilled water. Mix thoroughly, add 0.5 ml of the standard 4-aminophenazone solution, shake, and add 0.25 ml of potassium ferricyanide solution, and shake again for one minute. Similarly, pipette out 2, 4, 6, 8 and 12 ml of the standard solution and repeat the procedure. Plot a graph of optical density against concentration and check for linearity.

B-3.3 Measure the colour intensity with the help of spectrophotometer at 510 nm and compare the optical density of the sample with that of the standard and calculate the free phenol (as 2, 4-dichlorophenol) content.

ANNEX C

[Table 1, Item (iii)]

DETERMINATION OF LOSS ON DRYING**C-1 APPARATUS****C-1.1 Thermostatically Controlled Air-Oven****C-2 PROCEDURE**

C-2.1 Weigh accurately about 5 g of the sample into a dried, tared dish. Dry for 4 h in an air-oven at 105°C, cool in a desiccator, and weigh.

C-3 CALCULATION

C-3.1 Loss on drying, percent by mass $= \frac{100 (M - m)}{M}$

where

M = mass, in g, of material taken for the test; and

m = mass, in g, of the material after drying.

ANNEX D

[Table 1, Item (v)]

DETERMINATION OF TRIETHANOLAMINE INSOLUBLE MATTER**D-1 REAGENT****D-1.1 Triethanolamine Solution**

5 percent (m/v). Dissolve 5 g of triethanolamine salt in 100 ml of distilled water.

D-2 PROCEDURE

D-2.1 Dry a sintered glass crucible to constant mass at 105°C (alternatively, a Gooch crucible with prepared asbestos mat may be used). Weigh accurately about 5 g of the sample and dissolve in 80 ml of triethanolamine solution. Rinse with 50 ml of distilled water into a 250-ml beaker. Heat to boiling and stir until all

soluble material has dissolved. Filter hot through the crucible and wash three times with hot distilled water. Dry to constant mass at 105°C, cool, and weigh.

D-3 CALCULATION

D-3.1 Triethanolamine insoluble matter, percent by mass $= \frac{m \times 100}{M}$

where

m = mass, in g, of the dried residue; and

M = mass, in g, of the material taken for the test.

ANNEX E

[Table 1, Item (vi)]

DETERMINATION OF SULPHATED ASH**E-1 PROCEDURE**

E-1.1 Weigh accurately about 5 g of the sample into a tared silica crucible (minimum dimensions 51 mm × 51 mm). Moisten well with 95 percent ethanol or methanol and add 5 drops of concentrated sulphuric acid. Evaporate to dryness over a period of 1 to 2 h by heating gently, avoiding sputtering. Ignite until most of the carbon is burnt off. Allow to cool, add a few more drops of concentrated sulphuric acid, and re-ignite to constant mass. Express the mass of

residue as a percentage of the mass of the sample.

E-2 CALCULATION

E-2.1 Sulphated ash, percent by mass $= \frac{100 \times m}{M}$

where

m = mass, in g, of the sulphated ash; and

M = mass, in g, of the material taken for the test.

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Amendments Issued Since Publication

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